Tip-sample interactions on graphite studied in the thermal oscillation regime

Giovanna Malegori, Gabriele Ferrini *

Dipartimento di Matematica e Fisica, Università Cattolica del Sacro Cuore, I-25121 Brescia, Italy

**email: gabriele@dmf.unicatt.it webpage: http://www.dmf.unicatt.it/elphos/*

INTRODUCTION

Thermal noise is caused by random thermal excitations that result in positional fluctuations of the cantilever, thereby setting a lower limit on the force resolution in AFM measurements. However thermal noise, can also be utilized for measurement purposes.

From the modification of the thermal motion of the cantilever due to the tip-surface interaction forces it is possible to reconstruct the interaction potential and obtain information on various kind of surface forces.

In this work we will use three different approaches to exploit the information contained in the cantilever thermal motion and measure the force gradients near a surface a) measure the frequency shift of the flexural modes b) measure the Boltzmann distribution of the tip position Brownian motion c) measure the thermal mean-square displacement of the tip position [1].

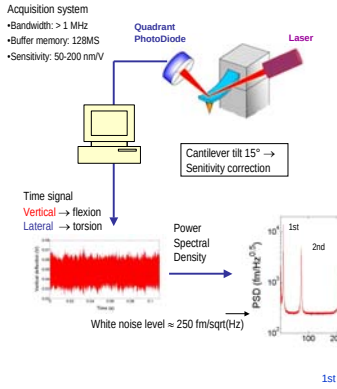
TIP-SAMPLE INTERACTION IN AIR

The jump to contact observed typically in NC-AFM under ambient conditions is not due to van der Waals forces, but is explained as a two-step process: first, when the tip is at a distance of about 2-3 nm, a liquid meniscus forms between tip and sample , and afterward, this meniscus pulls the tip onto the sample so that a mechanical contact between both is formed. It has been shown that this process depends on the relative humidity [12].

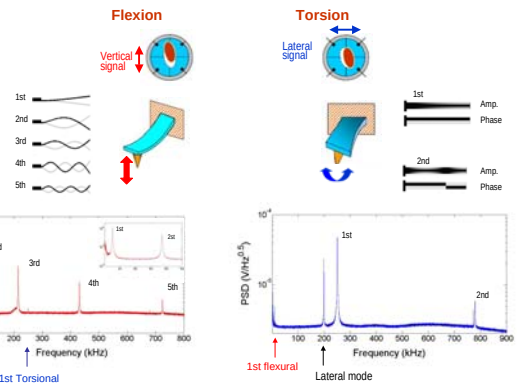
The distance dependence as well as the magnitude of the frequency shift observed in our experiments is in good agreement with a van der Waals interaction between tip and sample.

On the other hand, the reduction of the *Q*-factor has an extremely sharp distance dependence so that the underlying dissipation mechanism is very local, probably due to some interaction of the very end of the tip with the surface.

THERMAL OSCILLATIONS SETUP



FREE CANTILEVER



Free cantilever (non contact)

Flexural mode	1	2	3	4	5
f_n (kHz)	10.8	74.3	215	431	724
f_n / f_1 (exp.)		6.9	20	40	67
f_n / f_1 (th. [2])		6.27	17.55	34.39	56.84

supported cantilever (contact)

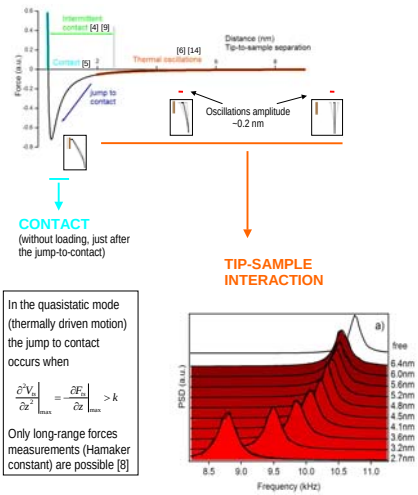
Flexural mode	1	2
f_n (kHz)	69.2	201
f_n / f_1 (exp.)	6.4	19
f_n / f_1 (th.) [2]	4.39	14.21

Free cantilever (non contact)

Torsional mode	1	2
f_n (kHz)	250	779
f_n / f_1 (exp.)	23	72
f_n / f_1 (th. [3])	24.71	74.13

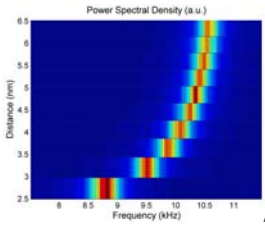
$$f_n^{\perp} = \alpha_n^2 \frac{h}{L^2}\sqrt{\frac{E}{12\rho}} \qquad \frac{f_n^{\perp}}{f_1^{\perp}} = \frac{(2n-1)^2\sqrt{6}}{\alpha_1^2}\sqrt{\frac{1}{1+\nu}\frac{L}{w}}$$

PROBING THE TIP-SAMPLE INTERACTION IN AIR



APPROACHING THE HOPG SURFACE

FREQUENCY SHIFT



Free cantilever resonance frequency $f_0 = \frac{1}{2\pi}\sqrt{\frac{k}{m^*}}$

k spring constant, *m** effective mass

The tip-sample interaction F_{ts} changes the resonance frequency (small oscillation amplitudes)

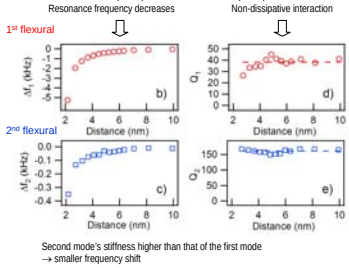
$$f_0 = \frac{1}{2\pi}\sqrt{\frac{k^*}{m^*}} \qquad k^* = k - \frac{\partial F_{ts}}{\partial z}$$

Frequency shift → tip-sample force gradient

$$f = f_0 + \Delta f \qquad \frac{\partial F_{ts}}{\partial z} = -2k\frac{\Delta f}{f_0}$$ [7] [8]

Attractive tip-sample force
Resonance frequency decreases

Tip-sample not in contact
Non-dissipative interaction



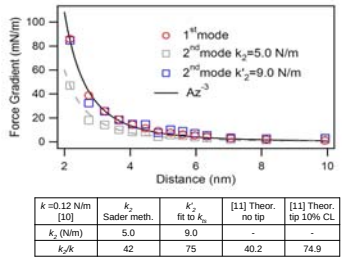
Long-range tip-sample force [2] $F_{ts} = -\frac{HR}{6(z-z_0)^2}$

H Hamaker constant, *R* tip radius (≈10 nm), *z*-z_0 tip-sample distance (piezo-tube position, z_0 surface position and cantilever static deflection $\Delta z = F_{ts}/k$)

Force gradient $\frac{\partial F_{ts}}{\partial z} = -\frac{HR}{3(z-z_0)^3}$

HR=3·10^{-27} J m

Second mode's stiffness higher than that of the first mode → smaller frequency shift



Sader method [10] is typically accurate only for the lower modes. In fact, the tip mass does not influence significantly the equivalent stiffness of the first eigenmode but can have dramatic effect on the equivalent stiffness of the higher eigenmodes. A tip mass that is 10% of the cantilever mass nearly doubles the second mode's equivalent stiffness [4] [11]

k =0.12 N/m [10]	k_2 Sader meth.	k'_2 fit to k_{ts}	[11] Theor. no tip	[11] Theor. tip 10% CL
k_2 (N/m)	5.0	9.0	-	-
k_2/k	42	75	40.2	74.9

POTENTIAL FROM BOLTZMANN DISTRIBUTION

Boltzmann probability distribution

$$p(s) = p_0 \exp(-V(s)/K_BT)$$

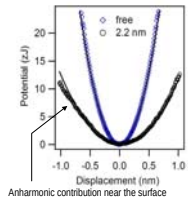
p(*s*) probability of observing the tip at a deflection *s* from the equilibrium position *z* or tip-surface distance *z*, p_0 normalization constant, *V*(*s*) position dependent potential

Inverting the relation gives the total potential

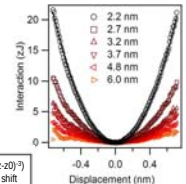
$$V(s) = -K_BT \ln\frac{p(s)}{p_0}$$

$$V(s,z) = V_C(s,z) + V_{ts}(s,z)$$

V_C cantilever potential, V_{ts} tip-sample interaction potential $\frac{\partial F_{ts}}{\partial z} = -\frac{\partial^2 V_{ts}}{\partial z^2}$ [13]

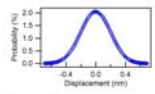


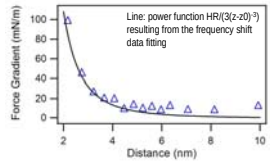
Anharmonic contribution near the surface



Interaction potential is parabolic→ mass-spring approximation correct

Gaussian probability distribution





MEAN SQUARE DISPLACEMENT

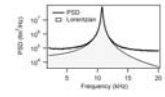
Equipartition Theorem $\frac{1}{2}K_BT = \frac{1}{2}k\langle s^2\rangle$

k free cantilever spring constant, $\langle\cdot\cdot\rangle$ cantilever mean square deflection

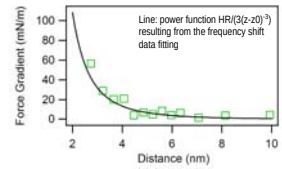
The first mode mean square virtual deflection $\langle s_1'^2\rangle$ and then the effective spring constant *k** are evaluated at different tip-sample distances.

$$k^* = 0.82\frac{K_BT}{\langle s_1'^2\rangle} \qquad \frac{\partial F_{ts}}{\partial z} = k - k^*$$ [14]

Frequency domain → thermal oscillations isolated from white noise background

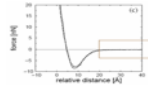


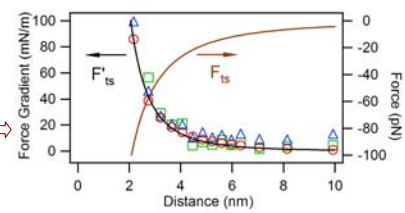
$\langle s_1'^2\rangle$ equals the integral of the power spectrum of the thermal fluctuation alone (Lorentzian function) → fit minus white-noise background



THERMAL OSCILLATIONS

Intermittent contact
Short-range forces [9]





1.Frequency shift o
2.Potential Δ
3.Mean square displacement □

Long-range interaction forces of the order of tens of piconewton. Tip-sample interaction force:

$F_{ts} = HR/(6(z-z0)^{-2})$

HR=3·10^{-27}Jm from the frequency shift data fitting.

FUTURE

- Investigation of other surfaces.
- Automate the spectroscopy
- Measurements in liquids
- Torsional mode analysis

The AFM used in this research has been developed by

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